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The Influence of Lipids on Self-Dispersion and on Ease of Dispersion of Milk Powder[a,b]

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Effective reconstitution of dry whole milk in water in preparation of a fluid product which simulates fresh homogenized milk depends upon rapid dispersion of the milk powder constituents. The role played by the lipid materials in self-dispersion and in dispersibility [d] has not been determined; yet their physical properties indicate their potential importance. For example, in the liquid state, certain lipids may alter the surface tension of the aqueous medium and the interfacial tension of powder particles. When some lipids are subjected to temperature change, which permits melting and solidification, different crystalline forms may occur.

Milk fat, a term used to represent all of the lipids present in milk, is chiefly a mixture of different glycerides. As a result, its transition from the solid to the liquid state is not sharp, but occurs over a temperature range. Rogers *et al (9)* report that the melting range of milk fat is 28°-36° C. (82.5°-96.8° F.).

In spray-dried whole milk powder the milk fat is distributed among the other powder materials in the form of globules. Globule size depends to a large extent on the processes employed in manufacturing. During reconstitution these globules are dispersed, along with the other powder constituents, and finally are resuspended as an emulsion in the serum of the reconstituted milk.

This research, a part of a program designed to improve the dispersibility of dry whole milk, was undertaken to investigate the influence of lipids on self-dispersion and on ease of dispersion by low energy stirring.

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[d] The term *dispersibility* has developed into common use, especially in connection with research and development of dry whole milk at the Quartermaster Food and Container Institute. Stegeman *(10)* used redispersion of dry whole milk to designate the act of preparing fluid milk from milk powder and dispersibility to imply a characteristic of a powder to redisperse. In this paper, dispersibility is used to designate ease of and completeness or extent of dispersion of milk powder in water with agitation to form a product which simulates fresh homogenized milk. The scope of this investigation did not include a study of completeness of dispersion.

REVIEW OF THE LITERATURE

The effect of heat on the solubility [e] of milk proteins has received the attention of many investigators *(4, 12)* but there is a lack of published information on the influence of milk fat and other lipids on dispersibility of dried milks. However, in connection with investigations on dispersibility, various methods for estimating and studying certain dispersing properties, (wettability, for example) were developed which reveal some of the approaches used in dispersibility studies.

Kleinert *(5)* described a method for measuring wettability, based on self-dispersion as determined by a turbidimetric technique. Later, Ashworth *(1)* proposed a wettability test in which the percent solids self-dispersed in 5 minutes under specific conditions was designated as percent wettable. Both of these investigators found that whole milk powders with good wetting properties, as determined by their techniques, were more soluble than those with poor wetting properties.

Nickerson *et al. (8)* estimated *wettability* in terms of the time required for milk powder particles to sink below the surface of water on which they were dusted, and *dispersibility* by noting the time required for particles to lose their identity when immersed in water at room temperature. The National Dairy Research Laboratories *(7)* developed a turbidimetric method for estimating self-dispersion and a shaking method for determining dispersibility. Cone and Ashworth *(3)* developed a new quantitative manual shaking method for determining the solubility of milk powders in which they attempted to simulate home kitchen conditions in respect to temperature, concentration, and agitation.

Tracy and Wilster *(11)* estimated reconstitutability by the appearance of undispersed material remaining after filtration of the reconstituted product through a black organdy pad having 74 meshes per inch; completeness of reconstitution was estimated (a) from the solubility index, (b) by microscopic examination of the milk film, and (c) by the appearance of the reconstituted milk after shaking one pint of the product in a quart bottle.

As pointed out in the preceding review, the characteristic property of milk powder to self-disperse in water, especially in reference to a time factor, has been associated by various workers with the wettability of the product. No established method for quantitative estimation of ease of dispersion of milk powder in water by low energy stirring is in evidence in the literature. Preliminary data obtained in connection with this study indicated, however, that the dispersing properties of whole milk powder are affected by the milk fat.

EXPERIMENTAL

Determination of self-dispersion. Apparatus: The apparatus for estimating self-dispersion consisted of a screening and testing device. The screening device was made by securely fastening a circle of screen 150 mm. in diameter to a ring, which was supported by a separate ringstand, 2 mm. above the top rim of the Buchner funnel of the testing apparatus. The function of the screen is to aid in manual distribution of the milk powder. The

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[e] Solubility has been used to designate the ability of milk powders to disperse in water to form a solution, suspension, or emulsion, which simulates the physical characteristics of natural milk; however, the term is generally recognized as a misnomer.

effectiveness is controlled, to a large extent, by the size of the screen, and a ¼-inch and 14 mesh screen were selected for use when testing whole and nonfat milk powders, respectively. The self-dispersion testing device consisted of an 82 mm. inside diameter Buchner funnel, a large stopcock, a coarse (40-60 micron) fritted glass Buchner type funnel (62 mm. I. D.), and a filtering flask. These parts were assembled as shown in Figure 1.

Procedures. (a) The powder was tempered (during at least 24 hours) to the desired temperature (the standard temperature of powder for testing was 70-75° F.). (b) Fifteen grams of tempered powder were distributed (by screening) over the surface of 90 ml. of water (standard temperature 70-75° F.) which was contained in the Buchner funnel of the testing apparatus. (c) After 3 minutes, the mixture was rapidly drawn into the filtering flask with the aid of vacuum (Ca 16 inches) and was diluted to 200 ml. in a volumetric flask. (d) Total solids were determined in a 20 ml. aliquot, and this weight times 10 represented the self-dispersion value for the sample under analysis.

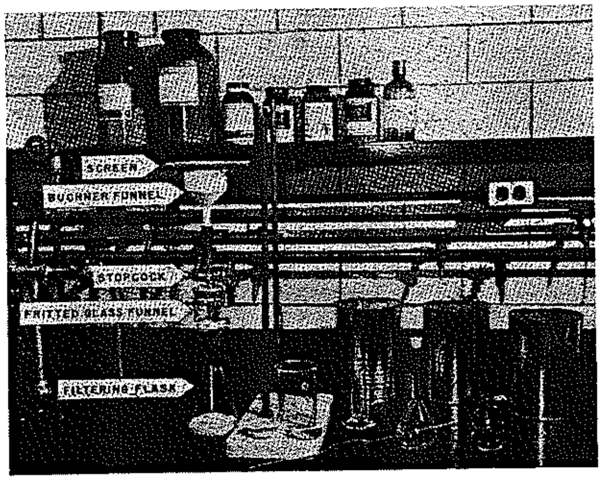

Figure 1. Apparatus for determining self-dispersion of milk powder.

Determination of ease of dispersion. Ease of dispersion by manual stirring was determined by the following procedures: (a) A representative sample of the powder under analysis was screened through a 14-mesh screen. (b) Fifteen grams of the screened powder were added to 90 ml. of water (standard temperature 70-75° F.) contained in a 250 ml. beaker. (c) The mixture was stirred for a designated stirring time with an ordinary teaspoon [f] and then filtered with the aid of vacuum (Ca 16 inches) through a coarse (40-60 micron) fritted glass Buchner type filtering funnel.[g] (d) The filtrate was diluted to 200 ml. and grams of total solids were determined in a 20 ml. aliquot. This value times 10 represented the disperson value for any one designated stirring time. (e) Ease of dispersion was estimated from the dispersion values resulting from 10, 20, 30 and 45 seconds stirring.

Total solid determinations. Twenty ml. of representative sample from the 200 ml. volume of diluted milk filtrate were transferred to a tared aluminum foil evaporating dish and were held in a well-ventilated oven at 70-75° C. until the solids appeared dry. Drying was completed in a convection current oven in accordance with time and temperature conditions for determination of total solids in milk as recommended in AOAC *(2)*.

[f] An attempt was made to disperse as much powder as possible, but care was exercised not to splash out of the beaker.

[g] Since difficulty in filtering was frequently encountered when testing high heat nonfat milk powder *(6)*, a 210 micron screen was used instead of the coarse fritted glass funnel when testing this type of powder.

Preparation of milk powder samples. The premium grade whole milk powders used in this study were made in accordance with MIL-M-1495A *(6)*. Those made in this Laboratory were dried with a Type N-3 Model N-25 G. Ind. turbulaire spray drier.[h] The inlet temperature was 350° F. and the outlet temperature, 185° F. The evaporating capacity with this temperature differential was approximately 10 pounds of water per hour. The atomizer pressure was about 60 p.s.i. with an estimated flow rate of air of 9 cubic feet per minute. The atomizer nozzle was a type N design IA 3.

RESULTS AND DISCUSSION

Influence of temperature of powder on self-dispersion. To obtain information on the effect of temperature of whole milk powder on self-dispersion, portions of a sample of premium grade product were adjusted to various temperatures between 5 and 140° F. then, self-dispersion in water at 75° F. was determined on each tempered sample at its respective temperature. The results appear in Table 1.

TABLE 1

Effect of temperature of dry whole milk powder on self-dispersion in water at 75° F.

Temp. of powder	Self-dispersion	Temp. of powder	Self-dispersion
° *F.*	*grams*	° *F.*	*grams*
5	0.846	80	0.684
15	0.816	85	0.762
35	0.786	95	0.893
40	0.806	100	6.337
50	0.793	110	7.776
60	0.695	120	8.320
70	0.613	140	8.786

These data (Table 1) show that self-dispersion varied widely over a range of temperatures. Between 5 and 70° F. a slight decrease in self-dispersion occurred and from 70 to 95° F. a slight increase occurred. However, between 95 and 100° F. (near the melting range of milk fat) a seven-fold increase in self-dispersion occurred. From 100 to 140° F. there was a marked but more gradual increase in self-dispersion of the dry whole milk.

In order to obtain additional data on the effect of temperature of dry whole milk on self-dispersion, five premium grade spray-dried powders, manufactured by different concerns, were each divided into two lots. One lot was tempered to 72° F. and the other to 120° F.; self-dispersion in water at 75° F. was determined on each sample at these respective temperatures. The whole milk powders which were tempered to 120° F. gave higher self-dispersion values than those tempered to 72° F. Four were 7 times higher, and one was 2.5 times higher.

Self-dispersion of dry whole milk may be affected by temperature fluctuations. To demonstrate the effect on self-dispersion of heating to 120° F., and cooling slowly to room temperature, 3 powders were tested at temperatures of 72° and 120° F. in water at 75° F. Subsequently, the samples which were heated to 120° F. were allowed to cool at room temperature for 1, 2, 3, 4, 5 and 20 hours. At the end of each cooling time, self-disper-

[h] Manufactured by the Western Precipitation Corporation, Los Angeles.

sion was again determined on each sample. The results are shown in Table 2.

These data demonstrate that slow cooling of whole milk powder from temperatures above the melting range of milk fat to temperatures below this temperature range decreases self-dispersion, and that the decreases occur over a period of several hours. Of interest was the fact that different self-dispersion values, as compared to the initial value at 72° F., were obtained after heating and cooling. This difference may be attributable to a different physical state of the newly solidified milk fat, and will require further study, particularly along lines of melting and solidification of mixed glycerides.

TABLE 2

Change in self-dispersion of dry whole milk due to heating and cooling water temperature 75° F.

Temp. of powder	Tempering time	Self-dispersion measurements		
		Sample L-2	Sample L-3	Sample L-4
° F.	*hours*	*grams*	*grams*	*grams*
72 (initial)	12	0.694	0.657	0.503
120	12	9.524	8.897	2.398
85	1	7.831	......	......
80	2	6.027	......	......
80	3	3.795	......	......
80	4	0.639	......	......
80	5	0.616	3.757	0.390
76	20	0.488	0.999	0.467

At powder temperatures below the melting range of milk fat, nonfat milk powders generally disperse more rapidly than whole milk powders. To demonstrate the effect of temperature of nonfat powder on self-dispersion in water at 75° F., samples of two spray-dried products were tempered to temperatures varying between 5° and 120° F.; and self-dispersion tests were made on each tempered sample. The results are presented in Table 3. These data show that increasing the temperature of nonfat milk powder increased self-dispersion; however, a large, sharp increase did not occur near the melting range of milk fat as occurred with whole milk powder.

TABLE 3

Effect of temperature of powder on self-dispersion of nonfat milk powder

Sample No. L-1 Fat content 1.25%		Sample No. L-2 Fat content 1.4%	
Temp. of non-fat powder	Self-dispersion in water at 75° F.	Temp. of non-fat powder	Self-dispersion in water at 75° F.
° F.	*grams*	*° F.*	*grams*
5	1.765	5	3.157
35	1.759	35	3.833
72	1.896	72	4.484
100	1.681	100	4.953
120	1.928	120	5.239

Influence of temperature of water on self-dispersion. To determine the effect of temperature of water on self-dispersion, whole and nonfat milk powders at a temperature of 72° F. were tested using water at various temperatures between 35 and 150° F.

The results (Table 4) show that self-dispersion of dry whole milk was little affected by temperature of water up to 90° F., but between 90 and 100° F. there occurred a large, sharp increase in self-dispersion, and between 100 and 140° F., a gradual increase in self-dispersion occurred.

Temperature of water had a different effect on self-dispersion of nonfat dry milk than it had on self-dispersion of dry whole milk. The nonfat powder demonstrated a gradual increase over the entire temperature range studied. In water at 150° F., self-dispersion of nonfat powder was comparable to that of whole milk powder in water at 100° F. The different self-dispersion values which were obtained at the various water temperatures in connection with whole and nonfat milk powder, indicate that temperature of water just above the melting range of milk fat acts to greatly increase self-dispersion of dry whole milk.

TABLE 4

Influence of temperature of water on self-dispersion of whole and nonfat milk powder at 72° F.

Temp. of water	Self-dispersion of milk powder	
	Whole	Nonfat
° F.	*grams*	*grams*
35	0.682	1.042
40	0.663	1.444
50	0.683	1.364
60	0.731	1.580
70	0.786	1.855
80	0.782	2.170
85	0.811	2.599
90	0.963	2.861
95	1.420	3.364
100	8.177	3.444
110	9.489	4.110
120	......	6.102
140	10.391	......
150	......	8.445

In order to further delineate the influence of lipids on self-dispersion, a 20% fat powder in which corn oil replaced milk fat was made. Samples of this product were tempered to various temperatures between 5° F. and 120° F. and self-dispersion was determined at each temperature in water at 75° F. The results appear in Table 5.

TABLE 5

The influence of corn oil on self-dispersion of milk powder in water at 75° F.

Temp. of powder	Self-dispersion	Temp. of powder	Self-dispersion
° F.	*grams*	*° F.*	*grams*
5	5.799	72	6.071
35	5.921	90	6.380
50	5.845	100	6.511
		120	6.492

At powder temperatures below the melting range of milk fat, higher self-dispersion values were obtained for corn oil powder than were obtained for normal dry whole milk, but at powder temperatures above the melting range of milk fat, normal dry whole milk gave higher self-dispersion values (Table 1) than corn oil powder (Table 5). The rapid self-dispersion of cold corn oil powder may be attributable to the presence of liquid corn oil at 75° F., the temperature of the water.

Effect of various levels of milk fat on self-dispersion. Milk powders, which varied in percent fat from 7.5 to 48.3, were prepared in this Laboratory. The amount of fat in the dried product was controlled by standardizing the skim milk concentrates with homogenized cream. The temperature on introduction to the dryer was 160° F. and drying was done in the Turbulaire dryer. Each powder was analyzed for percent fat and moisture; and was tested for self-dispersion at 72° F. in water at 75° F. The data obtained are presented in Table 6.

TABLE 6

Effect of various levels of milk fat on self-dispersion of milk powder

(Powder temperature, 72° F.; water temperature, 75° F.)

Sample No.	Self-dispersion	Percent moisture [1]	Percent fat [1]
	grams		
Nonfat control	4.559	2.3	1.4
F-1	1.578	2.0	7.4
F-2	1.448	2.2	9.8
F-3	0.955	2.2	27.0
F-4	0.527	2.3	34.9
F-5	0.351	1.6	48.3
M-1 [2]	0.909	2.0	26.2

[1] A.O.A.C. Procedures.
[2] Premium Grade Whole Milk Powder.

The effect of fat content on this characteristic dispersing property under these temperature conditions is shown by the large difference in self-dispersion between the nonfat control and Sample F-1. As the fat content of the powder was increased, self-dispersion decreased. In order to permit comparison with premium grade whole milk powder *(6)* the analysis of M-1, a commercial product, was also presented in Table 6. Samples F-3 and M-1 compared closely in self-dispersion and also in fat content.

It is probably coincidental that the two samples F-3 and M-1 (Table 6) gave nearly the same values for self-dispersion, even though the fat levels are approximately the same. For example, as is shown in Table 7, when 36 samples of premium grade dry whole milk were tested for self-dispersion, values between 0.303 and 1.209 were obtained. However, the majority of the samples were included in the ranges .500-.599, .600-.699, .700-.799, and .800-.899. The temperature of powder and water at

TABLE 7

Distribution of self-dispersion values of dry whole milk

Range in grams	Number of samples	Percent of total samples
1.000-plus	1	2.7
.900-.999	3	8.3
.800-.899	5	13.9
.700-.799	13	36.1
.600-.699	5	13.9
.500-.599	4	11.1
.400-.499	2	5.6
.300-.399	3	8.4
Total	36	100.0

the time of analysis was 72 and 75° F. respectively. These data indicate that factors other than the content of milk fat also affect self-dispersion.

Effect of temperature on ease of dispersion by stirring. Since temperature affected the self-dispersing characteristic property of dry whole milk, especially at the melting range of the milk fat, investigations were made as to the effect of temperature of dry whole milk on ease of dispersion by low energy manual stirring. To demonstrate this effect, 2 samples were taken from each of 3 lots of premium grade dry whole milk, and one sample from each lot was tempered to 72° F. and one to 120° F. Self-dispersion and ease of dispersion determinations were made in duplicate on each sample of powder at their respective temperatures using water at 75° F. The results which are average values appear in Table 8.

The expected increase in self-dispersion attributable to temperature of powder was obtained. Also, the effect of temperature of powder on ease of dispersion during

TABLE 8

Effect of temperature of dry whole milk on ease of dispersion in water at 75° F. by stirring

Sample no.	Temp. of powder	Self-dispersion in grams	Grams dispersed by stirring for: 10 sec.	20 sec.	30 sec.	45 sec.
D-2182	72° F.	0.752	5.675	9.862	10.423	11.486
	120	7.950	9.559	10.704	12.433	13.891
D-1383	72° F.	0.918	5.460	10.382	11.143	11.881
	120	7.660	9.718	10.919	12.257	13.151
D-1288	72° F.	1.221	5.082	11.142	12.037	12.661
	120	6.948	10.822	11.062	12.882	13.634

10 seconds stirring is clearly demonstrated. However, for the other stirring times, the temperature effect was not as evident by inspection of the data; therefore, analysis of variance was made on the results obtained for 20, 30, and 45 seconds agitation. The mean square deviation (duplicates) and the mean square variance attributable to temperature treatment together with their significant levels calculated through their F ratio appear in Table 9.

TABLE 9

Significant levels of variation on dispersion attributable to difference in temperatures of 72° F. and 120° F.

Stirring time	Mean square deviation (duplicates)	Mean square (variance attributable to temperature)	F ratio	Significant levels
seconds				%
20	.0965	0.563	5.83	5-10
30	.0695	5.250	76.00	.5
45	.0782	4.940	63.00	.5
Combined	.0805	9.238	115.00	.5

The variation in grams of powder dispersed attributable to temperature of powder is very definite when compared with variation due to error of replication except for 20 seconds stirring. For this stirring time the temperature differential was not clearly demonstrated with the available data.

The mean square (variance) attributable to time of stirring for all three times was 11.825 and that attributable to powder variation 2.551. The variance associated with length of stirring is comparable to the variance associated with temperature of powder, but both are considerably larger than the variance associated with the powders. However, all three sources of variance are significant at the 0.5 percent level when compared with the error variance.

The time-temperature interaction mean square is 0.751 which is significant at the 0.5 percent level with respect to the error mean square, and the differential thus varies significantly under different lengths of stirring time for the 20-45 second range. When considering only 30 and 45 seconds stirring times, the mean squares for temperature, time, and their interactions are, respectively 10.188, 3.726, and 0.003. The first two are definitely significant, but the last is clearly negligible. In this range of stirring time, the temperature effect is more important than the effect of stirring time and there is no evidence that the temperature differential differs for the two stirring times.

These data (Table 8) are important in that they demonstrate that whole milk powder tempered to 120° F. dispersed more easily by manual stirring in water at a temperature of 75° F. than does whole milk powder tempered to 72° F. These powder temperatures, 120° F. and 72° F., are, respectively, well above and below the melting range of milk fat.

SUMMARY AND CONCLUSIONS

Tempering dry whole milk to temperatures which cause melting of milk fat increased self-dispersion. Slow cooling of dry whole milk from 120° F. to 76° F. acted to decrease self-dispersion; however, four to five hours were required to obtain new values comparable to initial values. Milk powder in which 20% corn oil was substituted for the milk fat, demonstrated a gradual increase in self-dispersion concomitant with an increase in temperature of powder; however, a sharp increase in self-dispersion failed to occur at or near the melting range of milk fat.

Temperature of the water near the melting range of milk fat caused a large increase in self-dispersion of whole milk powder. As the milk fat content of milk powder was increased, self-dispersion decreased. The powders were tested at 72° F. in water at 75° F. Seventy-five percent of the self-dispersion values resulting from analysis of 36 samples of premium grade dry whole milk were within the following ranges: .500-.599; .600-.699; .700-.799; and .800-.899 g. When the temperature of the water was constant at 75° F., whole milk powder tempered to 120° F. dispersed more rapidly by manual stirring than powder tempered to 72° F.

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LITERATURE CITED

1. Ashworth, U. S. A method for measuring the wettability of milk powders. *J. Dairy Sci.*, 35, 481 (1952).
2. Association of Official Agricultural Chemists. *Official Methods of Analysis.* 7th ed., Washington, D. C., 1950.
3. Cone, J. F., and Ashworth, U. S. A new quantitative method for determining the solubility of milk powders. *J. Dairy Sci.*, 30, 463-472 (1947).
4. Howat, G. R., and Wright, N. C. Factors affecting the solubility of milk proteins. II. The influence of temperature of reconstitution on protein solubility. *J. Dairy Research*, 4, 265-272 (1933).
5. Kleinert, J. *Schweiz. Milchztg.*, 75, 397-98 (1949).
6. Milk, Dry; Whole and Nonfat Solids. Military Specification, 31 July 1952. Chicago Quartermaster Depot, Chicago, Illinois.
7. National Dairy Research Laboratory, Inc., Oakdale, Long Island, New York. Methods for measuring the dispersibility of whole milk powders. Report No. 419. 1951. (Unpublished.)
8. Nickerson, T. A., Coulter, S. T., and Jeness, R. Some properties of freeze-dried milk. *J. Dairy Sci.*, 35, 77-85 (1952).
9. Rogers, L. A., and Associates. *Fundamentals of Dairy Science.* 1928, The Chemical Catalog Company, Inc., New York, N. Y.
10. Stegeman, R. A. The dry milk industry. *Activities Report,* 1, 230-236 (1948). Published by the Research and Development Associates, Food and Container Institute, Inc., Chicago, Ill.
11. Tracy, P. H., and Wilster, G. H. Factors affecting reconstitutability of dry whole milk. File DD-4, Report No. 4, Committee on Food Research Technical Reports. Quartermaster Food and Container Institute, Research and Development Branch, Office of The Quartermaster General. 1946.
12. Wright, N. C. Factors affecting the solubility of milk powders. Part I. The effect of heat on the solubility of milk proteins. *J. Dairy Research*, 4, 123-141 (1932).